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“पुराने को छोड़ नये के तरफ”

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IS 231 (1957): Amyl Acetate [PCD 9: Organic Chemicals
Alcohols and Allied Products and Dye Intermediates]



“ज्ञान से एक नये भारत का निर्माण”

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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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REAFFIRMED

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(Reaffirmed 1995)

Indian Standard

REAFFIRMED

SPECIFICATION FOR AMYL ACETATE

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 5

June 1957

Indian Standard

SPECIFICATION FOR AMYL ACETATE

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AMENDMENT NO. 2 JUNE 1975
TO
IS : 231-1957 SPECIFICATION FOR AMYL ACETATE

Addendum

[*Page 2 (page 4 of the reprint)*, *clause 4.4*] — Add the following new clause after **4.4**:

4.5 Special Requirement — When the material is required for use in pyrotechnic industry, it shall also comply with the requirement of moisture content 0.3, percent, *Max* when tested by the method described in IS : 2362-1963 Determination of Water by the Karl Fischer Method.'

(CDC 2)

Reprography Unit, BIS, New Delhi, India

Indian Standard
SPECIFICATION FOR
AMYL ACETATE

FOREWORD

This Indian Standard was adopted by the Indian Standards Institution on 27 October 1950 as a tentative standard on the endorsement by the Chemical Division Council of the draft finalized on 25 August 1950 by the Fine Chemicals (Organic & Inorganic) Sectional Committee. In June 1957, this standard was issued as firm Indian Standard without any modification.

This standard prescribes the requirements for technical and pure grades of amyl acetate. In laying down the requirements of amyl acetate, technical, note has been taken of the fact that this material is supplied in the market from two sources viz, synthetic and, fusel oil.

In the preparation of this standard, considerable assistance was derived from the standards of the American Society for Testing Materials, the British Standards Institution and the Standards Association of Australia and the help derived from these sources is acknowledged.

This standard is intended chiefly to cover the technical provisions relating to the purchase of the material, and it does not include all the necessary provisions of a contract.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of test for Amyl Acetate, Technical and Pure. Amyl acetate, technical, is used mainly as a solvent by the paint, varnish and lacquer industry, and amyl acetate, pure, is used as a flavouring material in perfumes and in photo-engraving and as a laboratory reagent.

2. GRADES

2.1 There shall be two grades of amyl acetate, namely, Technical and Pure.

3. SAMPLING

3.1 Representative samples of the material shall be drawn as prescribed in Appendix A.

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4. REQUIREMENTS

4.1 Unless specified otherwise, chemicals of the purest grade shall be employed in the tests and distilled water shall be used where the use of water as a reagent is specified.

4.2 Description — Amyl acetate, technical or pure shall be a clear, colourless liquid, free from visible impurities.

4.3 Freedom from Water — No trace of opalescence shall be produced when 1 volume of the material is mixed, at 15°C, with 19 volumes of carbon disulphide.

4.4 The material shall also comply with the requirements in Table I, when tested according to the methods prescribed in the appendices referred to in column (5) of the table.

TABLE I REQUIREMENTS FOR AMYL ACETATE

SL No.	CHARACTERISTIC	REQUIREMENT FOR GRADE		METHOD OF TESTING (REF TO APPENDIX)
		Technical	Pure	
(1)	(2)	(3)	(4)	(5)
i)	Specific gravity at 20/20°C	0.857 to 0.870	0.868 to 0.876	B
ii)	Residue on evaporation, percent by weight, <i>Max</i>	0.02	0.01	C
iii)	Acidity, calculated as acetic acid, percent by weight, <i>Max</i>	0.03	0.01	D
iv)	Ester content, percent by weight, <i>Min</i>	85	97	E
v)	Range of distillation	100 ml of amyl acetate shall yield on distillation at 760 mm pressure not less than 85 ml between 125 and 145°C	100 ml of amyl acetate shall yield on distillation at 760 mm pressure not less than 95 ml between 125 and 145°C	F

5. PACKING AND MARKING

5.1 The material shall be supplied, in accordance with the marking and delivery instructions given by the purchaser, in leak-proof containers approved by the purchaser.

5.2 Each container shall be marked with the name of the manufacturer, the grade and weight of the material in the container, recognized trade mark, if any, and the month and year of manufacture.

APPENDIX A

(*Clause 3.1*)

SAMPLING OF AMYL ACETATE

1. GENERAL REQUIREMENTS OF SAMPLING

1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

1.1 Samples shall be taken in a protected area with good ventilation.

1.2 The sampling instrument shall be clean and dry when used.

1.3 The samples, the material being sampled, the sampling instrument and the containers for samples shall be protected from adventitious contamination.

1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking or stirring or rolling or by any other suitable means so as to bring all portions into uniform distribution.

1.5 The samples shall be placed in clean, dry and air-tight glass or metal containers on which the material has no action.

1.6 The sample containers shall be of such a size that they are almost, but not completely, filled by the sample.

1.7 Each sample container shall be sealed air-tight with a suitable stopper after filling and marked with the manufacturer's name or trade-mark, grade of the material, the month and year of manufacture of the material, the batch number (if available) and other details of sampling, such as the date of sampling, sampler's name, etc.

1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

2. SAMPLING INSTRUMENT

2.1 Sampling Tube — It is made of thick glass or metal on which the material has no action and is 20 to 40 mm in diameter and 400 to 800 mm in length (*see Fig. 1*).

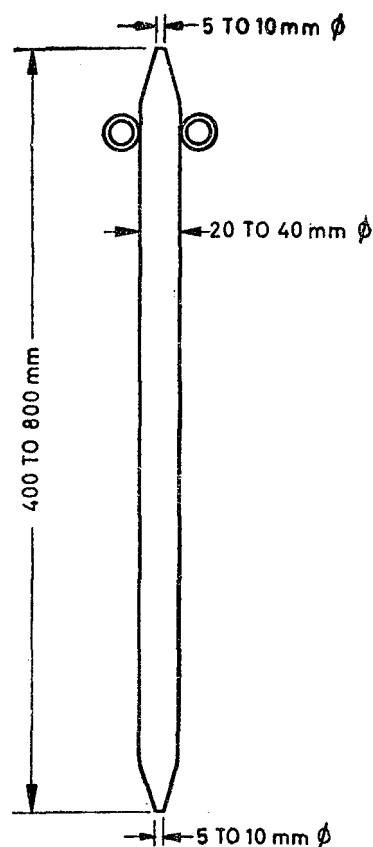


FIG. 1 SAMPLING TUBE

The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For drawing sample, the apparatus is first closed at the top with the thumb or a stopper and lowered till a desired depth is reached. It is then opened for a short time to admit the material at the desired depth and finally closed and withdrawn.

2.1.1 For small containers, the size of the sampling tube may be altered suitably.

3. SCALE OF SAMPLING

3.1 Lot — In any consignment, all the containers of the same grade and size, drawn from the same batch of manufacture, shall constitute a lot. If a consignment consists of containers of different grades, or of different batches of manufacture or of different sizes, the containers belonging to the same grade, batch and size shall be grouped together and each such group shall constitute a separate lot.

3.2 For ascertaining the conformity of the material in a lot to the requirements of this specification, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose from a lot shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table II.

TABLE II SCALE OF SAMPLING

LOT SIZE	NO. OF CONTAINERS TO BE SELECTED
N	n
(1)	(2)
Up to 3	*
4 „ 15	3
16 „ 40	4
41 „ 65	5
66 „ 110	7
111 „ 180	8
181 „ 300	9
301 and above	10

*In case the lot consists of 3 or less than 3 containers, the number of containers to be selected and the method of judging the conformity of the lot to the specification requirements shall be as agreed to between the purchaser and the supplier.

3.3 Containers shall be selected at random from the lot and, in order to ensure randomness of selection, a random number table as agreed to between the purchaser and the supplier shall be used. In case such a table is not available, the following procedure is recommended for use:

Arrange all the containers in the lot in a systematic manner and starting from any container count them as 1, 2. . . up to r and so on, where r is the integral part of N/n (N being the lot size and n , the number of containers to be selected). Every r th container thus counted shall be withdrawn to constitute the sample.

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4. PREPARATION OF TEST SAMPLES

4.1 From each of the containers selected according to **3.3**, a representative portion of the material, sufficient for carrying out the tests specified under **REQUIREMENTS** shall be drawn with the help of the sampling tube (*see 2.1*).

4.2 Out of these portions, equal quantity of material shall be taken and mixed thoroughly to form a composite sample, not less than 1 500 ml. The composite test sample shall be divided into 3 equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

4.3 The composite samples shall be transferred to containers of 600 ml capacity and shall be sealed and marked with full identification particulars given in **1.7**.

4.4 The referee test sample shall also bear the seal of both the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier, to be used in the case of any dispute between the two.

5. CRITERIA FOR CONFORMITY

5.1 The lot shall be declared as conforming to the standard, if the test results of the composite test sample satisfy the requirements prescribed under **REQUIREMENTS**. Otherwise the lot shall be rejected.

A P P E N D I X B

[*Table I, Item (i)*]

DETERMINATION OF SPECIFIC GRAVITY

I. DEFINITION

1.1 For the purposes of this standard the specific gravity of the material is the ratio of the weight of a given volume of the material to that of an equal volume of distilled water, the temperature of both the material and the water being 20°C.

2. APPARATUS

2.1 A specific gravity bottle of about 50 ml capacity.

3. PROCEDURE

3.1 Clean, dry and weigh the bottle and the cap. Fill with water, introduce the cap and keep it at a temperature of 20°C for 15 minutes. Suck off the water with a bit of filter paper, till the level reaches the graduation mark, and weigh again. Empty the bottle, clean and dry it, and repeat the operations with the material.

3.2 Calculation and Report — The temperature of testing shall be specified in the report.

$$\text{Specific gravity} = \frac{c - a}{b - a}$$

where

a = weight of the dry specific gravity bottle.

b = weight of the specific gravity bottle filled with distilled water, and

c = weight of the specific gravity bottle filled with the material.

APPENDIX C

[Table I, Item (ii)]

DETERMINATION OF RESIDUE ON EVAPORATION

1. PROCEDURE

1.1 Evaporate 100 ml of the material, accurately measured into a tared nickel, silica or hard glass basin of about 75 mm diameter and 20 mm deep. Dry the residue for one hour at $150 \pm 2^\circ\text{C}$. Cool the residue in a desiccator and weigh.

1.2 Using the specific gravity of the material determined under Appendix B, calculate the residue on evaporation as percentage of the weight of the material taken.

$$\text{Residue on evaporation, percent} = \frac{B - A}{S}$$

where

A = weight of empty basin,

B = weight of basin after evaporation and drying, and

S = specific gravity of the material.

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A P P E N D I X D

[*Table I, Item (iii)*]

DETERMINATION OF ACIDITY

1. REAGENTS

1.1 Phenolphthalein Indicator — Dissolve 0.5 g of phenolphthalein in 100 ml of 95 percent ethyl alcohol and make the solution faintly pink by adding sodium hydroxide solution.

1.2 Standard Sodium Hydroxide Solution — approximately 0.01 N.

2. PROCEDURE

2.1 Mix 20 ml of the ester, delivered from a pipette, with 20 ml of neutral alcohol and titrate the mixture with standard sodium hydroxide solution using 0.5 ml of phenolphthalein indicator.

3. CALCULATION

3.1 Using the specific gravity determined under Appendix B, calculate the acidity as percentage by weight on the basis that 1 ml of 0.01 N sodium hydroxide solution is equivalent to 0.0006 g of acetic acid.

A P P E N D I X E

[*Table I, Item (iv)*]

DETERMINATION OF ESTER CONTENT

1. REAGENTS

1.1 Standard Alcoholic Sodium Hydroxide — approximately 0.5 N.

1.2 Standard Hydrochloric Acid — approximately 0.5 N.

1.3 Phenolphthalein Indicator — prepared as in 1.1 of Appendix D.

2. PROCEDURE

2.1 Weigh accurately 1 to 1.5 g of the ester in a small, stoppered, tared tube, and transfer the tube with its contents to a flask containing 50 ml of

alcoholic sodium hydroxide solution. Fit a water-cooled reflux condenser to the flask and heat the latter for one hour over a boiling water bath. At the end of the heating period, rinse the condenser with well-boiled and cooled distilled water. Cool, remove the flask and titrate the contents with standard hydrochloric acid, using 0.5 ml of phenolphthalein indicator.

2.2 Carry out a blank determination, using all the materials excepting the ester, under similar conditions and at the same time.

3. CALCULATION

3.1 From the difference in the volumes of standard sodium hydroxide required in the titrations with and without the material, and using the factor that 1 ml of 0.5 N alcoholic sodium hydroxide is equivalent to 0.065 of amyl acetate, calculate the weight of the ester for 100 g of the material.

APPENDIX F

[Table I, Item (v)]

DETERMINATION OF RANGE OF DISTILLATION

1. APPARATUS

1.1 Distillation Flask — of the shape and dimensions given in Fig 2. Fix the flask in the vertical position by means of a clamp at the extreme upper end of the neck.

1.2 Thermometer — so fitted in the flask that the bottom of the capillary is level with the lower edge of the side-tube joint and the immersion mark is level with the top of the cork.

1.2.1 The recommended dimensions, tolerances and graduations of the thermometer shall be as follow:

Range	90 to 160°C
Graduation	0.2°C
Longer lines at each	1°C
Fully figured at each	5°C
Immersion	100 mm
Overall length	315 to 325 mm
Length of main scale, <i>not less than</i>	170 mm
Bulb length, <i>not greater than</i>	15 mm
Stem diameter	5 to 6.5 mm

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Distance from the bottom of bulb to bottom of main scale	100 to 125 mm
Distance from the bottom of bulb to top of contraction chamber, <i>not greater than</i>	25 mm
Maximum error	0.8°C
Maximum error in an interval	0.8/10°C

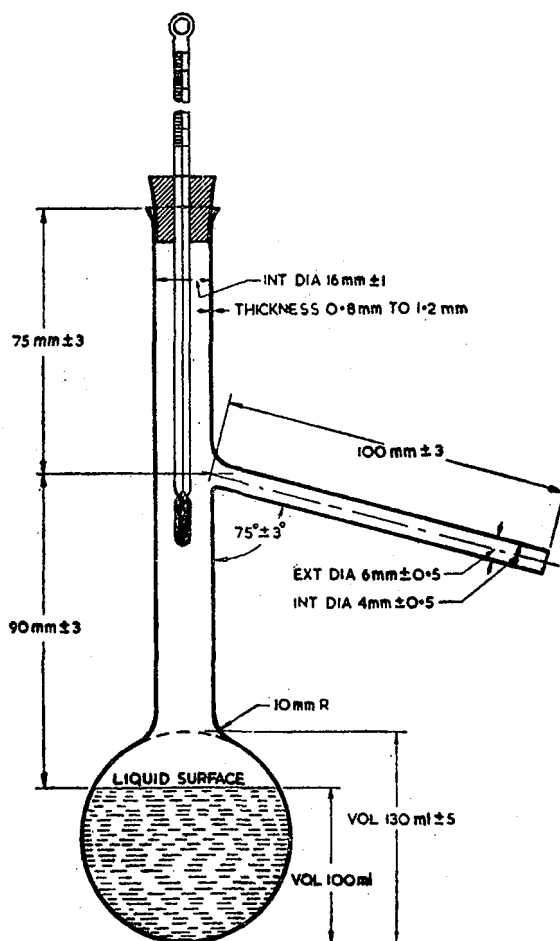


FIG. 2 DISTILLATION FLASK

1.2.2 The thermometer shall bear a certificate of the National Physical Laboratory of India or any other institution authorized by the Government of India to issue such a certificate.

1.3 Rectangular Draught Screen — made of 0.710 mm thick sheet metal, with the dimensions shown in Fig. 3 and open at the top and bottom, fitted with hard asbestos or silica board, with a central hole 100 mm in diameter. It shall comply with the following requirements.

1.3.1 In each of the two narrow sides of the draught screen there shall be two circular holes, each 25 mm in diameter, and in each of the four sides of the draught screen there shall be three holes with their centres 25 mm above the base of the draught screen. These holes shall occupy the positions shown in Fig. 3, the diameter of each of the holes centrally situated in the longer sides shall be 25 mm and, of the remaining ten holes, shall be 12.5 mm. At the middle of each of the wider sides a vertical slot with the dimensions shown in Fig. 3 shall be cut downwards from the top of the screen.

1.3.2 A sheet of hard asbestos, 6 mm in thickness, and having a central circular hole, 50 mm in diameter, shall be supported horizontally in the screen and shall fit closely to the sides of the screen to ensure that hot gases from the source of heat do not come in contact with the sides or neck of the flask. The supports for this asbestos sheet may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners.

1.3.3 In one of the narrow sides of the screen a door shall be provided having the dimensions and position as shown in Fig. 3. In each of the narrow sides of the screen a mica window shall be placed centrally, with the bottom of the window on a level with the top of the asbestos shelf. The dimensions and position of the windows are shown in Fig. 3.

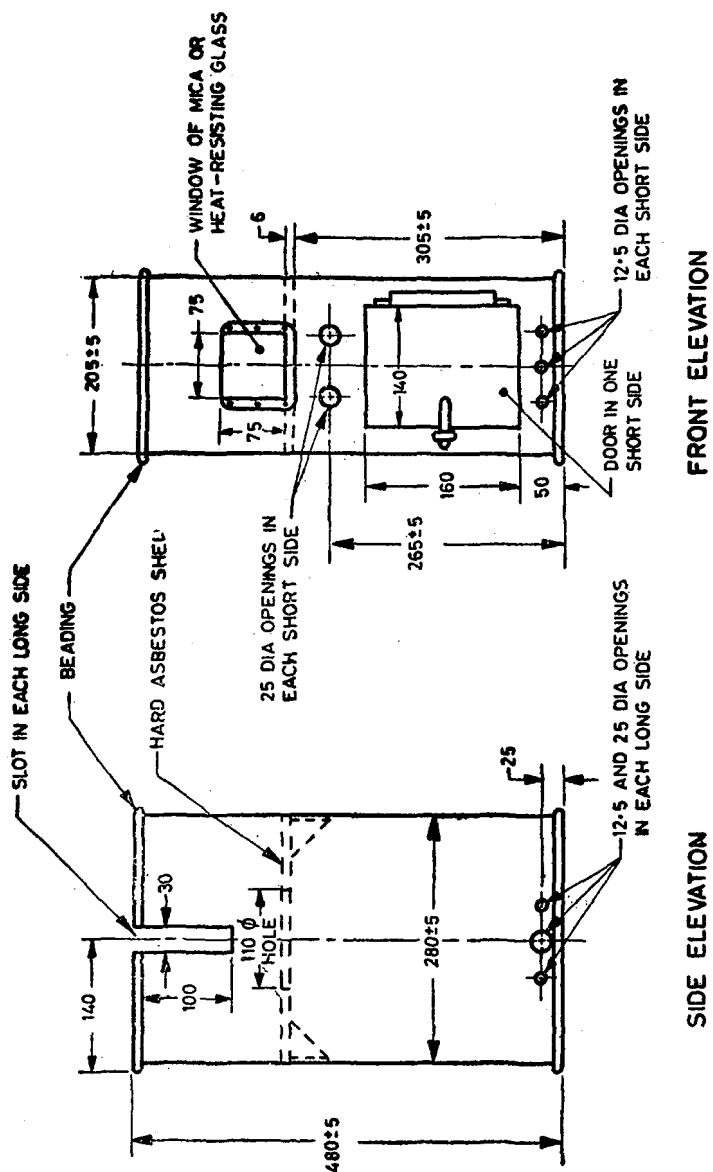
1.4 Liebig Condenser — with the bent end made of good quality resistance glass, with a wall thickness of 1 to 1.5 mm and conforming to the shape and dimensions given in Fig. 4.

1.4.1 *Alternatively*, the bent portion may be substituted by an adapter fitted externally in such a manner that the distillate does not come in contact with the cork.

1.5 100 ml Crow Receiver (Fig. 5) or Graduated Cylinder — with millilitre marks running halfway round the circumference, with 5 millilitre marks running three-quarters-way round, and with 10 millilitre marks running all round the circumference, and numbered.

1.6 Bunsen Burner — giving an entirely non-smoky flame and provided with an adjustable screw clip with the help of which the flame can be lowered or raised according to requirement.

1.7 Half-Second Pendulum — for measuring the rate of distillation.



All dimensions in millimetres.

FIG. 3 DRAUGHT SCREEN

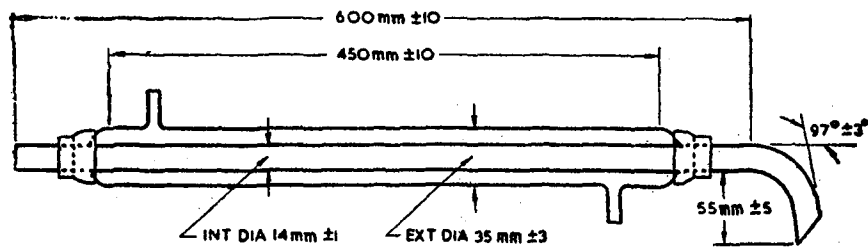


FIG. 4 LIEBIG CONDENSER

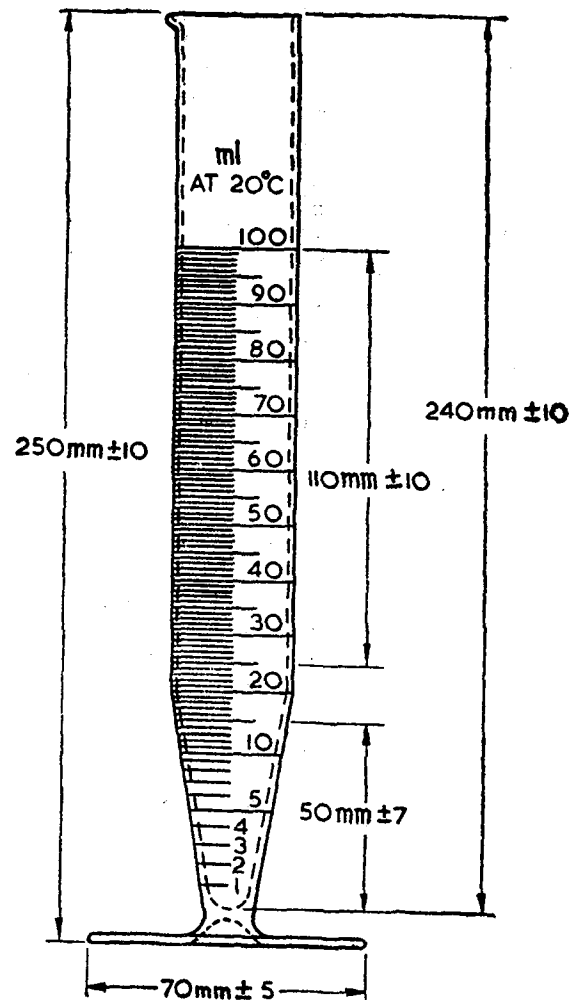


FIG. 5 100 ml CROW RECEIVER

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2. PROCEDURE

2.1 Assemble the apparatus as shown in Fig. 6. Measure 100 ml of the material at laboratory temperature into the 100 ml Crow receiver and transfer it to the distillation flask, the contents of the receiver being allowed to drain for fifteen seconds into the flask. Add a fragment (about 2 mm cube) of porous pot or other suitable inert material to prevent bumping, connect the flask to the condenser and insert the thermometer. Pass an adequate supply of cooling water through the condenser. To receive the distillate, use the Crow receiver in which the sample was measured, without rinsing or drying. Heat the flask slowly, especially after ebullition has begun, in order that the mercury column of the thermometer may become fully expanded before the first drop of distillate falls into the receiver, care being taken that the total period of this preliminary heating shall be not less than five nor greater than ten minutes. Place the receiver so that the condensate will flow down its side. Continue the distillation at the rate of 4 to 5 ml per minute (about 2 drops per second). Read the volume of distillate in the receiver when the thermometer indicates each of the specified distillation temperatures, the temperatures on the thermometer scale being corrected as specified under 3. The difference between the volumes so recorded is the percentage by volume distilling between the specified temperatures at 760 mm pressure.

3. CORRECTION OF THE THERMOMETER READING

3.0 The following corrections shall be applied before starting distillation.

3.1 Error of Scale — In all thermometer reading, make the corrections as indicated on the certificate of the instrument.

3.2 Correction for Barometric Pressure.— If the barometric pressure prevailing during the determination is normal, namely 760 mm of Hg, no correction need be applied to the specified temperature, and the thermometer scale as corrected under 3.1 may be used as such. If the prevailing barometric pressure deviates from 760 mm, the specified temperature shall be corrected as follows and used on the thermometer scale as corrected under 3.1.

3.2.1 For every 10 mm above 760 mm add 0.43°C to the specified temperature and for every 10 mm below 760 mm subtract 0.43°C from the specified temperature. These corrections shall be applied in proportion at the above rate for any prevailing barometric pressure and are valid down to a pressure of 700 mm of Hg.

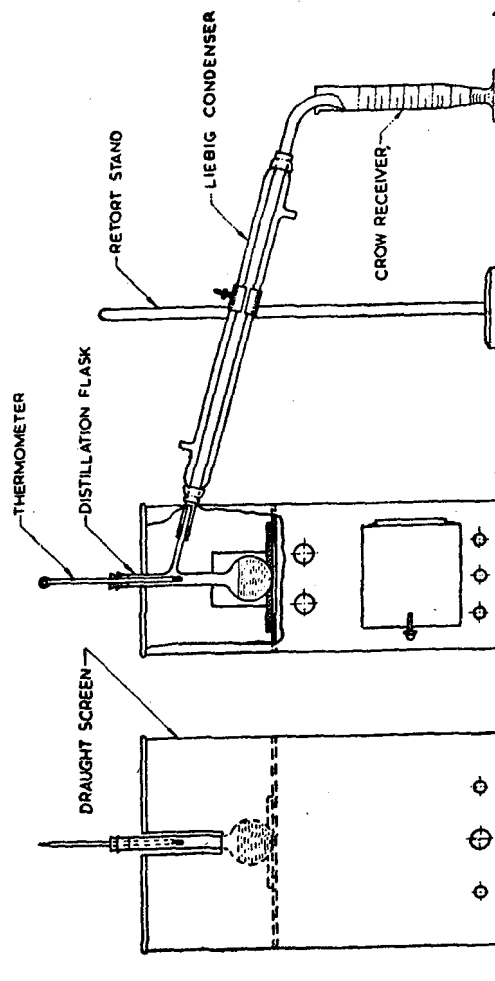


FIG. 6 ASSEMBLY OF APPARATUS

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002
Telephones: 323 0131, 323 3375, 323 9402 **Fax:** + 91 011 3234062, 3239399, 3239382
E - mail: bis@vsnl.com. **Internet:** http://wwwdel.vsnl.net.in/bis.org

Central Laboratory:

Plot No. 20/9, Site IV, Sahibabad Industrial Area, Sahibabad 201010	Telephone 77 00 32
---	------------------------------

Regional Offices:

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	323 76 17
*Eastern : 1/14 CIT Scheme VII, V.I.P. Road, Kankurgachi, CALCUTTA 700054	337 86 62
Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022	60 38 43
Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113	235 23 15
†Western : Manakalaya, E9, MIDC, Behind Marol Telephone Exchange, Andheri (East), MUMBAI 400093	832 92 95

Branch Offices:

'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMEDABAD 380001	550 13 48
‡Peenya Industrial Area, 1st Stage, Bangalore-Tumkur Road, BANGALORE 560058	839 49 55
Commercial-cum-Office Complex, Opp. Dushera Maidan, E-5 Arera Colony, Bittan Market, BHOPAL 462016	72 34 52
62/63, Ganga Nagar, Unit VI, BHUBANESWAR 751001	40 36 27
Kalai Kathir Building, 670 Avinashi Road, COIMBATORE 641018	21 01 41
Plot No. 43, Sector 16 A, Mathura Road, FARIDABAD 121001	28 88 01
Savitri Complex, 116 G.T. Road, GHAZIABAD 201001	71 19 98
53/5 Ward No.29, R.G. Barua Road, 5th By-lane, GUWAHATI 781003	54 11 37
5-8-56C, L.N. Gupta Marg, Nampally Station Road, HYDERABAD 500001	320 10 84
E-52, Chitrangan Marg, C- Scheme, JAIPUR 302001	37 38 79
117/418 B, Sarvodaya Nagar, KANPUR 208005	21 68 76
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